This Page Is Inserted by IFW Operations and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

As rescanning documents will not correct images, please do-not report the images to the Image Problem Mailbox.

(12) UK Patent Application (19) GB (11) 2 299 024 (13) A

(43) Date of A Publication 25.09.1996

- (21) Application No 9605535.5
- (22) Date of Filing 15.03.1996
- (30) Priority Data
 - (31) 9506039
- (32) 24.03.1995
- (33) GB

(71) Applicant(s) Unilever Plc

Unitever Pic

(Incorporated in the United Kingdom)

Unilever House, Blackfriars, LONDON, EC4P 4BQ, United Kingdom

- (72) Inventor(s)
 - Martinho Correia
- (74) Agent and/or Address for Service
 Naoise Edward Gordon
 Unilever Plc, Colworth House, Sharnbrook, BEDFORD,
 MK44 1LQ, United Kingdom

- (51) INT CL⁶
 A61K 7/32 7/48
- (52) UK CL (Edition O)
 A5B BFG B825 B826 B832
- (56) Documents Cited EP 0515195 A1 US 5219560 A WPI Abstract Acc. No. 86-194758/30 & JP610129187 A
- (58) Field of Search

 UK CL (Edition O) A5B BFG

 INT CL⁶ A61K 7/32 7/48

 ONLINE: CAS ONLINE; DIALOG/WPI, CLAIMS, JAPIO

(54) Antiperspirant containing an alkyl ester siloxane

(57) An underarm composition suitable for topical application to the human skin, such as a cream, comprising an antiperspirant and/or deodorant active agent, a cosmetic vehicle, and an alkyl ester siloxane wax, preferably behenic acid silicone wax, of general formula (I);

(Me₃Si)O(SiMe₂O)_aSi(Me)(SiMe₃)(CH₂)_b-O-COR

wherein

a is an integer from 0 to 2 b is an integer from 2 to 4

Me is CH₃

R is straight or branched chain alkyl from C₁₀H₂₁ to C₃₀H₆₁

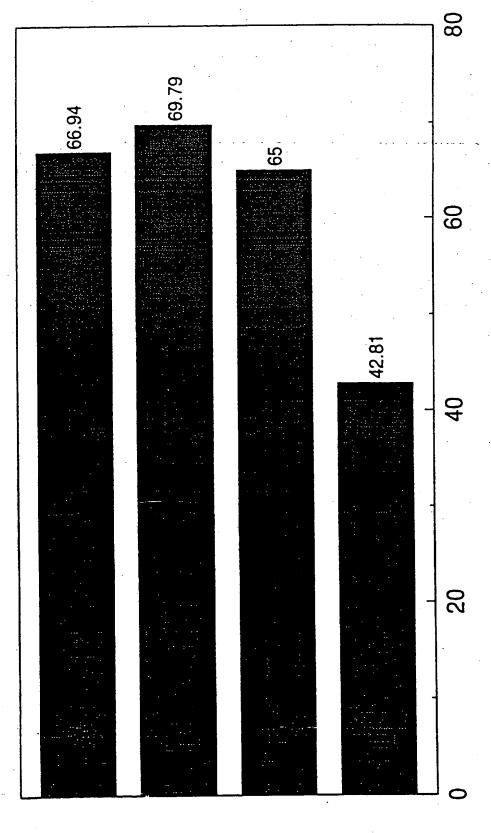
The composition may be an aerosol, cream or stick.

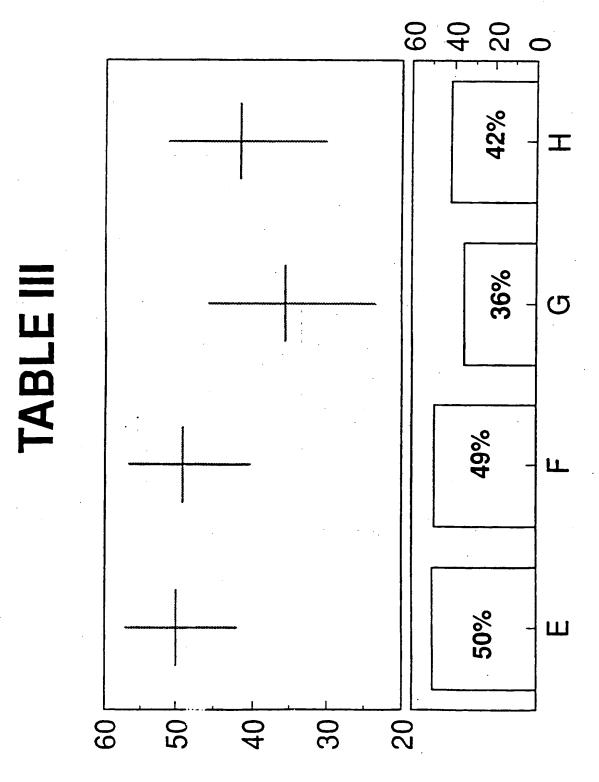
TABLE

| | | <u> </u> | | | | , | . — | | | |
|-------------------|--------------|-----------|------------------------|-------------|-------------|-----------------------|----------|----------------|-------------|-----------|
| | 24.0 | ı | 15.0 | 1 | 5.0 | 50.0 | 5.0 | ı | 1 | 1.0 |
| O | 24.0 | 10.0 | 1 | 4.0 | 0.9 | 43.0 | • | 10.0 | 2.0 | 1.0 |
| മ | 24.0 | 15.0 | 1 | • | 0.9 | 54.2 | 1 | 1 | 1 | 0.8 |
| Α | 24.0 | 15.0 | . 1 | - | 6.0 | 54.0 | • | 1 | 1 | 1.0 |
| Ingredient [%wt.] | AZAG Q5-7167 | Mirasil B | Alkyl Silicone Wax (5) | Aerosil 200 | Talc | Volatile Silicone (4) | Glycerol | Polydecene (6) | Propylene | Fragrance |

(5) DC X2-1834, ex Dow Corning (6) Silkflo 364NF

Comparison of Sensory Properties of A,B,C,D TABLE II





10

15

20

25

30

35

UNDERARM COMPOSITIONS

This invention relates to novel underarm compositions which contain a silicone wax material. More particularly, it refers to antiperspirant and deodorant compositions for topical application to the human skin, containing silicone ester wax compounds.

It is known for underarm compositions for topical application to contain non volatile silicone liquids. These compositions often contain polyorganosiloxanes, which impart desirable characteristics to the composition. In particular, they provide emolliency to the composition, and may also provide (in particular with antiperspirant compositions) a masking effect of any solids present in the composition once the volatile components have evaporated, but without causing any serious reduction in efficacy of the compositions.

Representative of this art is EP 28,853 (Procter & Gamble).

It is also known to include certain categories of alkyl siloxane waxes into underarm compositions. For example, in EP 549,223 (Dow Corning), there is described underarm formulations containing certain long chain alkyl silicone wax compositions. These waxes are alleged to provide the formulations with desirable characteristics, including modified hardness, reduced whitening, improved feel, and compatibility with other ingredients.

We have discovered that a further category of silicone wax compounds may advantageously be incorporated into underarm compositions for topical application. Such compositions may have superior properties to known compositions, in particular with regard to improved feel on application of the composition to the skin. Moreover, the compositions do not inhibit antiperspirant or deodorant efficacy.

10

20

25

30

Thus, according to a first aspect, the invention provides an underarm composition suitable for topical application to the human skin, comprising an antiperspirant and/or deodorant agent, a cosmetic vehicle, and an acyl ester siloxane wax of general formula (I);

 $(Me_3Si)0(SiMe_20)_aSi(Me)(CH_2)_b-0-COR(SiMe_3)$

wherein a is an integer from 0 to 2 b is an integer from 2 to 4 Me is CH_3 R is straight or branched chain alkyl from $C_{10}H_{21}$ to $C_{30}H_{61}$

Preferably, R is straight chain alkyl $C_{19}H_{39}$. In addition, preferably a is 1 and b is 3.

It has been found that antiperspirant and/or deodorant agent compositions containing the acyl ester siloxane wax described may have particularly good sensory properties, including good feel on the skin on application. The inclusion of such acyl ester siloxane waxes may also allow the compositions to be formulated without some of the other ingredients which are typically found in antiperspirant or deodorant compositions, or at least to reduce the amount of them required to produce an acceptable product. Such other ingredients, the amounts of which may be reduced or eliminated in the composition, include additional structurants, such as hydrophobic clays or silicas, volatile silicones, such as cyclomethicones, or non volatile silicones, such as dimethicones.

In addition, when the topical composition is in the form of an antiperspirant composition, the presence of the acyl ester siloxane wax may act to partially or totally mask any

10

15

20

25

30

35

whiteness caused on the skin by the presence of the antiperspirant active salt.

Traditional underarm products often contain other ingredients to impart various characteristics to the final product.

These extra ingredients will also depend on the product form, which can typically be a stick, roll on lotion, cream, or propellant driven aerosol, to name a few.

For example, if the product according to the invention is an antiperspirant composition, the composition will typically include an antiperspirant salt. Typically used antiperspirant salts include inorganic and organic salts of aluminium and zirconium, and mixtures thereof. Particularly preferred are the aluminium, zirconium salts of aluminium halides, aluminium hydroxhalides, aluminium zirconium salts and mixtures thereof. Particularly preferred antiperspirant salts include so called activated aluminium chlorohydrate compounds, as for example are described in EP 6,739 (Unilever NV et al). Further antiperspirant active materials are described in EP 28,853, the contents of both of these applications being incorporated by reference.

For use in antiperspirant compositions, preferably the antiperspirant salt is present at a level of 5-30% by weight of the composition.

Compositions according to the invention may also preferably contain a volatile silicone material. Typically used volatile silicone materials include cyclic or linear polydimethylsiloxanes. Preferred cyclic polydimethylsiloxanes have from 3-6 silicone atoms, and a viscosity of less than 10 mm²s⁻¹(cSt) at 25°C. Preferred linear polydimethylsiloxanes have from 3-9 silicone atoms, and a viscosity of less than 5 mm²s⁻¹(cSt) at 25°C. Preferred

10

15

20

25

polydimethylsiloxanes are available for example from Dow Corning Corporation, under such names as Dow Corning 344 and 345. Preferably, if used in compositions according to the invention, the volatile silicone is present at a level of 30-60% by weight, more preferably 40-60% by weight.

The silicone wax used in compositions according to the invention is an acyl ester silicone wax. The inclusion of this wax in the formulation may impart some emolliency to the composition; it may therefore be possible to do without, or reduce, the amount of emollient materials, such as volatile or non-volatile silicones, which are conventionally included in such compositions. In addition, the acyl ester waxes when used in compositions according to the invention may impart some structure to the composition, thereby avoiding the need for other structurants, or at least reducing the amounts that are needed.

A particularly preferred wax for use in compositions according to the invention is a behenic acid silicone wax, which in general structure I has a value for a of 1 and for b of 3, and in addition has R as $C_{19}H_{39}$. Such a wax is available from Rhone Poulenc under the tradename Mirasil-B 71649 wax.

Preferably, such acyl ester waxes are present in the composition at a level of 1-30%, more preferably 2-25% by weight of the composition.

Compositions according to the invention may take any convenient product form, including lotion, stick, cream, pump spray, or propellant driven aerosol.

10

15

20

25

30

A convenient form is a solid stick, which may conveniently be contained in a suitable holder or dispenser to enable it to be applied.

An alternative form is a lotion, which may conveniently be used in a roll ball dispenser, fitted with a ball valve, which may be applied to the skin in a conventional manner. A further possible composition would be a liquid suitable for dispensing from a finger operated pump spray or hand operated pump spray, which can deliver without the use of propellant gases a fine spray to the skin.

The composition can also take the form of a cream which is suitable or adapted for topical application to the skin.

A further possible product form is in the form of a liquid, which is suitable for application from a propellant driven aerosol by use of a suitable propellant, examples of which are well known in the art. It should be noted that, when the composition is in the form of a liquid to be applied from a propellant driven aerosol, the percentages of the components of the composition referred to, in particular the amount of alkyl ester siloxane wax in the composition, refer to the so-called "concentrate" composition, before its dilution with propellant and dosage into the aerosol container.

Propellant driven aerosol compositions according to the invention will be packaged with an aerosol propellant. The propellant gas can be any liquefiable gas known in the art for use in aerosol containers. Examples of suitable propellants include trichlorofluoromethane, trichlorotrifluorethane, monochlorodifluoromethane, difluoroethane, propane, butane, isobutane, used singly or in combination. In such product forms, the composition

10

according to the invention may typically comprise 5-45%, and the propellant 55-95% by weight of the total composition.

Particularly preferred product forms of compositions according to the invention are creams, lotions, and sticks.

Compositions according to the invention may also include various optional ingredients, which will depend on the product type and form of the cosmetic product. Such optional ingredients include;

- non-volatile silicones, such as dimethyl cyclosiloxanes or polydimethyl siloxane, eg, DOW CORNING 200 fluids;
- deoperfumes and deodorant compounds which can act as antimicrobial agents, and generally provide the composition with a deodorant active capability;
- skin feel improvers, such as talc and finely divided polyethylene, an example of which is Acumist B18;
 - humectants, such as polyols, for example glycerol;
 - hydrocarbon emollients, such as polydecenes;
 - hydrophobic oils such as liquid paraffins, or isopropyl myristate;
- thickening and gelling agents, such as clays, for example, Bentone 38, silicas, for example, Aerosil 200, organic waxes, such as castor wax, beeswax, or paraffin wax, silicone waxes, stear, l alcohol, fatty alcohols having 14-24 carbon atoms, fatty acids having 16-36 carbon atoms, and polyethylene;

25

- 7 -

- perfumes;
- preservatives and antioxidants;
- 5 skin benefit agents, such as allantoin;
 - colours;
- other cosmetic adjuncts conventionally employed in stick, roll-on lotion, liquid spray, propellant driven aerosol, and cream products.

Preferably the composition is essentially anhydrous; that is it contains less than about 1% by weight of water.

Such optional ingredients conveniently make up the balance of the composition, and as such may typically comprise 10-95% by weight of the composition.

20 <u>Examples</u>

The invention will now be described by way of example only.

Example 1

25

15

The following examples may be prepared by conventional techniques, and all provide satisfactory compositions according to the invention.

30

Suspension Cream Antiperspirant

| | Component | <u>&w/w</u> |
|---|------------------------------|-----------------|
| | AZAG(1) | 24.0 |
| 5 | Behenic Acid Silicone Wax(2) | 15.0 |
| | Cyclomethicone | 55.0 |
| | Talc | 6.0 |

- 10 (1) Q5-7167, activated aluminium zirconium glycinate, ex Summit.
 - (2) Mirasil-B 71649 wax, ex Rhone Poulenc.

Cream Antiperspirant

15

20

| Component | <u>&w/w</u> |
|------------------------------|-----------------|
| AZAG(1) | 24.0 |
| Polyethylene | 14.0 |
| Behenic Acid Silicone Wax(2) | 12.0 |
| Cyclomethicone | 50.0 |

Cream Antiperspirant

| | Component | <u>%w/w</u> |
|----|---------------------------|-------------|
| 25 | AZAG(1) | 26.0 |
| • | Silica(3) | 2.5 |
| | Propylene Carbonate | 0.5 |
| | Cyclomethicone | 63.0 |
| | Behenic Acid Silicone Wax | 8.0 |

30

(3) Aerosil 200, ex Degussa.

Aerosol Antiperspirant/Deodorant

| | Ingredient | 8 wt. |
|---|-----------------------------------|-------|
| | Activated aluminium chlorohydrate | 8.00 |
| 5 | Bentone 38 | 4.00 |
| | Behenic Acid Silicone Wax (2) | 6.00 |
| | Fragrance | 4.00 |
| | Cyclomethicone | 78.00 |

10 Base (concentrate) used as 25% by weight of formulation with 75% hydrocarbon propellant.

High Efficacy Aerosol Antiperspirant

| 15 | <u>Ingredient</u> | g wt. |
|----|-----------------------------------|-------|
| | Activated aluminium chlorohydrate | 40.00 |
| | Bentone 38 | 4.00 |
| | Behenic Acid Silicone Wax (2) | 6.00 |
| | Fragrance | 4.00 |
| 20 | Cyclomethicone | 46.00 |

Base (concentrate) used as 25% by weight of formulation with 75% hydrocarbon propellant.

25 <u>Aerosol Antiperspirant</u>

| | Ingredient | <u>% wt.</u> |
|----|-------------------------------|--------------|
| | Aluminium chlorohydrate | 30.00 |
| | Bentone 38 | 8.00 |
| 30 | Behenic Acid Silicone Wax (2) | 25.00 |
| | Fragrance | 10.00 |
| | Isopropyl Myristate | 2.00 |
| | Cyclomethicone | 25.00 |

15

Base (concentrate) used as 23% by weight of formulation with 77% hydrocarbon propellant.

1.00

Aerosol Antiperspirant

Perfume

| | Ingredient | : 8 wt. |
|----|-----------------------------------|---------|
| | Activated aluminium chlorohydrate | 8.00 |
| | Bentone 38 | 2.00 |
| | Ethanol | 7.50 |
| 10 | Urea | 0.50 |
| | Behenic Acid Silicone Wax (2) | 2.00 |

Pentane 36.00 CAP30 hydrocarbon propellant 43.00

High Efficacy Roll-On Antiperspirant

| | Ingredient | <u>% wt.</u> |
|----|-----------------------------------|--------------|
| | Activated aluminium zirconium | 25.00 |
| 20 | tetrachlorohydrex glycine complex | |
| | Behenic Acid Silicone Wax (2) | 6.00 |
| | Volatile Silicone | 57.50 |
| | Clay | 3.00 |
| | Polyethylene | 6.00 |
| 25 | Propylene carbonate | 1.00 |
| | Fumed silica | 0.50 |
| | Fragrance | 1.00 |

Roll-On Antiperspirant

| | Ingredient | & Wt. |
|----|--|-------|
| | Zirconium aluminium pentachlorohydrate | 15.00 |
| 5 | Clay | 2.00 |
| | Ethanol | 2.50 |
| | Fragrance | 0.50 |
| | Behenic Acid Silicone Wax (2) | 5.00 |
| | Cyclomethicone | 75.00 |
| 10 | | |
| | High Efficacy Stick Antiperspirant | |
| | Ingredient | % Wt. |
| | Activated aluminium zirconium | 25.00 |
| 15 | tetrachlorohydrex glycerine complex | |
| | Behenic Acid Silicone Wax (2) | 4.00 |
| | Cyclomethicone pentamer blend | 43.00 |
| | Stearyl alcohol | 20.00 |
| | Hydrogenated castor oil | 4.00 |
| 20 | PPG-14 Butyl ether | 2.00 |
| | Magnesium aluminium silicate | 1.00 |
| | Perfume | 1.00 |
| | Unfragranced Stick Antiperspirant | |
| 25 | | |
| | Ingredient | % Wt. |
| | Activated aluminium zirconium | 17.00 |
| | tetrachlorohydrex glycerine complex | |
| | Behenic Acid Silicone Wax (2) | 14.00 |
| 30 | Cyclomethicone pentamer-tetramer blend | 53.00 |
| | Hydrogenated castor oil | 4.00 |
| | Glyceryl tribehenate | 2.00 |
| | PPG-8 distearate | 1.00 |
| | Stearyl alcohol | 9.00 |
| 35 | | |

35

25

Stick Antiperspirant

| | Ingredient | 용 wt. |
|----|--------------------------------|-------|
| | Aluminium chlorohydrate powder | 20.00 |
| 5 | Behenic Acid Silicone Wax (2) | 20.00 |
| | Cyclomethicone tetramer blend | 39.50 |
| | Behenyl alcohol | 14.00 |
| | Silicone dioxide | 1.00 |
| | C18-C36 triglycerides | 4.00 |
| 10 | Fragrance | 1.50 |

Example 2

The following cream formulation was prepared;

| | Component | <u>% w/w</u> |
|----|--------------------------------------|--------------|
| | AZAG(1) Behenic Acid Silicone Wax(2) | 24.0 |
| 20 | Talc Suprafino | 15.0 6.0 |
| | Perfume | 1.0 |
| | Volatile Silicone(4) | 54.0 |

(4) DC Q2/1465, ex Dow Corning.

This composition is a cream, with good stability and sensory properties.

In contrast, a similar formulation containing DC580

(Stearoxytrimthyl silane and stearyl alcohol), in place of the Mirasil B71649, did not thicken well, or provide a cream with good properties.

10

15

Example 3 Comparative Example

Cream formulations A,B,C,D as described in Table I were prepared. Formulations A,B and C contained an acyl silicone wax while Formulation D contained an alkyl silicone wax.

The formulations were compared for sensory properties using a "smoothness to touch" attribute. A Quantitative Descriptive Analysis test was employed on a sensory profiling panel of users. The users applied a set target dose to their axilla and scored the attribute on a scale of 0-100.

The results are tabulated in Table II. As can be seen from Table II formulations A,B and C containing an acyl ester wax achieved significantly higher scores than Formulation D containing an alkyl silicone wax.

Example 4. Comparative Example

20 The following formulations were examined:

| | | | 8 w/w | | |
|----|-------------------------------|----|-------|----|----|
| | Ingredient | E | F | G | Н |
| | Behenic Acid Silicone Wax (2) | 15 | 15 | - | - |
| 25 | AZAG | 24 | 24 | 24 | 24 |
| | Volatile Silicone (4) | 54 | 52 | 66 | 72 |
| | Talc | 6 | 6 | - | - |
| | Fragrance | 1 | 1 | _ | - |
| | Silica | - | 2 | - | 4 |
| 30 | Castorwax | _ | - | 10 | _ |

Efficacy of each of the formulations was compared in standard tests carried out on users under Hot Room conditions.

The results are shown in tabular form in Table III. As shown in the Table Formulation E and F based on the acyl ester wax exhibited higher efficacy than formulations G and H based on castorwax and volatile silicone respectively. Moreover, the formulations E and F show that the use of an acyl ester wax is not detrimental to efficacy when compared with a simple silica/silicone based composition.

Example 5. Comparative Example

10

5

Formulations A and C were compared with an ethanol control roll-on for efficacy in terms of deodorancy. Scores were allotted by users for malodour after 24 hours. The results are given below:

15

Formulation Malodour Score (Control)

A 1.42 (2.46)

C 1.35 (2.35)

20

Accordingly, the acyl silicone ester products had lower malodour scores than the control thereby indicating a lack of interference in deodorant efficacy.

15

25

30

35

CLAIMS

 An underarm composition suitable for topical application to the human skin, comprising an antiperspirant and/or deodorant agent, a cosmetic vehicle, and an acyl ester siloxane wax of general formula (I);

 $(Me_3Si)0(SiMe_20)_aSi(Me)(CH_2)_b-0-C0R(SiMe_3)$

- wherein a is an integer from 0 to 2 b is an integer from 2 to 4 Me is CH_3 R is straight or branched chain alkyl from $C_{10}H_{21}$ to $C_{30}H_{61}$
 - 2. An underarm composition according to claim 1, where the composition is an antiperspirant composition containing 5-30% by weight of an antiperspirant agent.
- 20 3. An underarm composition according to claim 1 or claim 2, wherein R is straight chain alkyl C_{19} H_{39} , a is 1 and b is 3.
 - 4. An underarm composition according to any of the preceding claims, wherein the composition additionally comprises 30-60% by weight of a volatile silicone.
 - 5. An underarm composition according to any of the preceding claims, wherein the acyl ester siloxane wax is present at a level of 1-30% by weight of the composition.
 - 6. An underarm composition according to any of the preceding claims, wherein the composition is in the form of a cream, lotion or stick.





Application No:

GB 9605535.5

Claims searched:

. C. CCCCOOF QE

Examiner:

Dr J Houlihan

Date of search:

19 June 1996

Patents Act 1977 Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.O): A5B BFG

Int Cl (Ed.6): A61K 7/32 7/48

Other: ONLINE: CAS ONLINE; DIALOG/ WPI, JAPIO, CLAIMS

Documents considered to be relevant:

| Category | Identity of document and relevant passage | | |
|----------|--|--|--|
| A | EP 0515195 A1 (GENERAL ELECTRICAL CO.) page 2 lines 3- 16; page 3 lines 43-55; page 9 lines 33-39 | | |
| A | US 5219560 (KOBAYASHI KOSE LTD.) column 1 lines 9-23; column 2 lines 35-47; Example 3 | | |
| A | WPI Abstract Acc. No. 86-194758/30 & JP610129187 A (NISSHIN OIL MILLS) See abstract | | |

Member of the same patent family

- A Document indicating technological background and/or state of the art.
- P Document published on or after the declared priority date but before the filing date of this invention.
- E Patent document published on or after, but with priority date earlier than, the filing date of this application.

X Document indicating lack of novelty or inventive step

Document indicating lack of inventive step if combined with one or more other documents of same category.